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With amended claims and statement.

(54) Title: DESINFECTING AGENT

(57) Abstract

Method of making a solution by mixing a hydride of a metal with a molar equivalent of a strong acid wherein the metal and anion of the acid form an essentially insoluble salt that can be removed by filtration.

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DISINFECTING AGENT

FIELD OF INVENTION

This invention applies, and relates, to aqueous solutions for killing infections microbes and particularly to an aqueous solution having a large concentration of oxonium ions that is a strong bactericide.

PRIOR ART AND INFORMATION DISCLOSURE

Many different substances can be classified as disinfectants. Chemical agents acting as disinfectants include strong acids and bases. The mode of action include one of five general types: oxidation, hydrolysis, modification of cell membrane permeability, mechanical disruption, chemical union.

A number of sulfur containing pharmaceutical compounds have been disclosed for use as bactericides. For example, U.S. Patent 4,006,251 to Taylor et al discloses Thiocarbamylsulfenamide compounds.

U.S. Patent 5,084,449 discloses bis (4-aminophenyl-sulfonees.

An apparatus (MicroWater™ distributed by Optimum Health Institute. San Mateo, Cal.) has been disclosed. The device produces two kinds of water with different redox potentials, one with a high reduction potential (referred to as "alkaline MicroWater") and one with a high oxidation potential (referred to as "acid MicroWater").

The acid MicroWater has been found to have commercially viable bactericidal properties when used in the lowest pH range (2.5) attainable (reported) for this solution. It is believed that the active molecule is the "hydronium" ion having the structure:

H+
H-O-H

It would be desirable to produce a solution of substantially exclusively oxonium ions having a pH less than 2.5 that would kill microbes on contact with the solution while not having a deleterious effect on human tissue since such a solution would be expected to have a stronger bactericidal effect than presently available solutions of oxonium ions.

The desire to express germicidal activity of various agents quantitatively has led to the development of numerous procedures, most of which are based on a phenol coefficient method developed by Rideal and Walker in 1906. The method involves culturing two batches of selected microbes for a period of time, one batch is disposed in a "standard" bactericide and the other batch is disposed in the "text" bactericide. A quantitative expression of effectiveness of the test bacteria may be expressed as a percent of the number of bacteria killed by test bactericide compared to the standard bactericide. Standard tests are outlined under ASTM guidelines.

SUMMARY

In view of the above, it is an object of this invention to provide a stable microbicidal solution of substantially exclusively oxonium ions that is more effective than presently available solutions of oxonium ions having a pH of greater than 2.5.

It is an additional object that the solution be sufficiently stable for viable commercial purposes.

This invention is directed toward a method of making the solution which includes mixing a hydride of a metal with a molar equivalent of a strong acid wherein the metal and anion of the acid form an essentially insoluble salt that can be removed from the solution by filtration leaving an aqueous solution having concentration of Ca of not more than 2500 ppm and a concentration of sulfate ions of not more than 2500 ppm and a pH less than 2.5.

In a preferred embodiment, the acid is sulfuric acid and the metal hydride is calcium hydride.

DESCRIPTION OF THE FIGURE

FIGURE 1 shows a method for making the invention.

FIGURE 2 shows Fig. 1 in greater detail.

DETAILED DESCRIPTION

Turning now to a description of the flow diagrams.

FLOW DIAGRAM 1

1. select acid and metal hydride
2. add acid to water
3. add metal hydride to acid solution
4. filter the solution

FLOW DIAGRAM 2

1. add one mole of concentrated sulfuric acid to water.
2. add 1 gram equivalent weight of analytic grade CaH_2 to the solution.
3. stir the solution.
4. pass solution through 10 micron filter.
5. allow solution to digest.
6. filter solution through a 2 micron filter.

Flow Diagram 1 is a flow diagram for one method of making the solution of this invention.

In step 1, a strong acid is selected together with the hydride of a metal wherein the metal and the anion of the acid form a substantially insoluble salt.

In step 2 the acid is added to water forming an acid having an appropriate concentration.

In step 3 the metal hydride is added to the acid solution in a gram equivalent amount equal to the acid leading to the precipitation of the metal salt of the acid.

In step 4, the metal salt is filtered from the solution.

Flow Diagram 2 shows Flow Diagram 1 in greater detail where the method in which the metal is calcium and the acid is sulfuric.

In step 1, one mole of concentrated analytic grade of sulfuric acid is added to triple distilled water.

In step 2, slowly add 1 gram equivalent weight of analytic grade CaH_2 to the solution.

In step 3, slowly stir until the reaction is complete producing a new solution.

In step 4, pass the new solution through a 10 micron filter, removing all particles of $CaSO_4$ larger than 10 microns.

In step 5, allow the solution to digest for 10 to 12 hours.

In step 6, filter the solution through an 11 micron filter.

To illustrate the invention by way of example, a sample of the invention was prepared in accordance with Flow Diagram 2. Triply distilled water was used and the resultant test sample was found to contain less than 2500 ppm of sulfate and less than 2500 ppm of calcium. The pH was 2.0. Bactericidal properties of the solution were evaluated by an independent laboratory, BioVir Laboratories, Inc. using the procedure ASTM E1153-87 which is hereby incorporated as reference into this specification. The method was modified in the following manner:

1. 22 mm^2 coverslips were used for the inocula step (step 3.2).

2. Only 0.6 mL of ARS-I, prepared according to the technique described in U.S. Patent No. 5,830,838 (namely, mixing $Ca(OH)_2$ and KOH with equivalent sulfuric acid in water then passing the solution through a 10 micron filter), was used on the coverslips.

3. Trypticase Soy Broth (TSB) pH 10 was employed as pH neutralizer.

4. 10 mL of TSB pH 10 with 0.6 mL of the test sample resulted in a final pH 6.7.

5. The test organisms were Staphylcoccous aureus and Enterobacter aerogenes

6. Sterile petri dishes were substituted for sterile glass jars.

The results of the test are presented in Table I.

TABLE I

Organism/ Test	Expected (cfu/mL)	Observed	Log	% reduction
S.aureus Control	NA	50,000	NA	NA
S.aureus A	50,000	<1	>4.5	99.995
S.aureus B	50,000	<1	>4.5	99.995
S.aureus C	50,000	90	2.7	99.9
E.aerogenes Ctrl	NA	53,000	NA	NA
E.aerogenes A	53,000	<1	>4.7	99.995
E.aerogenes B	53,000	<1	>4.7	99.995
E.aerogenes C	53,000	<1	>4.7	99.995

Conclusion of the testing lab (Bio-Vir):

According to these test results, the test sample has demonstrated a capability of a 99.9 ->99.99% bactericidal effect within five minutes.

The pH of the test solution resulting from preparing the test solution in accordance with fig. 2 was measured to be 2.0 compared 2.7 which was the lowest value reported using the electrolysis method discussed in the BACKGROUND of the specification. The lower the attainable pH, it would be expected that the greater would be the bactericidal power of the product.

The action of the solution of this invention has been tested many times in this laboratory at pH 2.0 and it has been found that there is no reaction whatsoever with periods of fifteen minutes exposure.

While not wanting to be bound by theory, it is believed that the resultant solution consists essentially of hydronium ions in view of the purity of the water, (being less than 2500 ppm of either Ca or Sulfate) in spite of a pH of 2.0.

The solution prepared according to the techniques of this invention is apparently an oxidizing acid that can withdraw electrons from bacteria and kill them. The solution contained in sufficient concentration (2.7 or lower) can be used to clean hands and utensils, meats, vegetables, fruit and sterilize cutting boards and wounds. Tests have shown that solutions of this type can be used effectively to treat athlete's foot, burns, insect bites and wounds. It is excellent for cleansing and household use. It has bleaching ability. It disinfects and sterilizes yet is harmless to the skin. It is an astringent. It tightens skin.

A major advantage of the present invention over the prior art is the ease and economy of preparing the solution. The present method is a chemical method whereas the competing process is an electrophoretic method. The competing process has the major disadvantage that the equipment is relatively expensive and difficult to maintain due to such factors as fouling of filter membranes by metal ions that are initially in the water. Furthermore, the achievable pH of the electrolytic process (reported 2.7) is not as low as can be achieved with the present invention. Another major advantage is that the solution of the present invention maintains a pH of less than 2.5 for longer than 48 hours (actually months) compared to the electrolytic

process of the prior art where pH remains at 2.7 for only a few hours.

Variations and modifications of the invention may be suggested by reading the specification which are within the scope of the invention. For example, other metal hydrides may be considered along with acids forming insoluble metal salts with the metal. These would include not only metals of the 2A Group but also certain precious metals.

These would include:

Barium hydride and sulfuric acid;

Beryllium hydride and sulfuric acid.

We therefore wish to define the scope of our invention by the appended claims.

WHAT IS CLAIMED IS:

1. A method for preparing a solution having disinfectant properties which comprises the steps of:

(a) selecting a metal hydride and an acid having an anion capable of forming an insoluble salt and said metal of said metal hydride;

(b) mixing a gram molar quantity of said acid in water;

(c) stirring into said acid in water an equivalent of said gram molar quantity of said metal hydride;

(d) passing the resultant solution of step (c) through a filter to remove precipitates of salt formed by said metal and said acid.

2. The method of claim 1 wherein said step (a) includes the step:

selecting said metal hydride to be calcium hydride and acid to be sulfuric acid.

3. A solution consisting essentially of less than 2500 parts per million of calcium sulfate, and a pH of less than 2.5

4. A solution consisting essentially of less than 2500 parts per million of calcium sulfate, and a pH of less than 2.5 wherein pH of less than 2.5 maintained for longer than 48 hours.

5. A method for preparing a solution having a pH of less than 2.5, and less than 2500 parts per million of calcium sulfate which comprises the steps of:

(a) forming a solution of one mole H_2SO_4per one liter of water;

(b) stirring into said solution of step (a) one Gram Equivalent Weight of C_aH_2 per one mole of H_2SO_4;

(c) filtering the solution of step (b) through an eleven micron filter;

(d) allowing the solution of step (c) to digest for at least 10 hours;

(e) filtering the solution of step (d) through a two micron filter.

AMENDED CLAIMS

[received by the International Bureau on 7 February 2000 (07.02.00); original claims 1 and 5 amended; original claim 3 cancelled; remaining claims unchanged (2 pages)]

1. A method for preparing a solution having disinfectant properties, said solution consisting essentially of less than 2500 parts per million of calcium sulfate and a pH of less than 2.5, which method comprises the steps of:

(a) selecting a metal hydride and an acid having an anion capable of forming an insoluble salt and said metal of said metal hydride;

(b) mixing a gram molar quantity of said acid in water;

(c) stirring into said acid in water an equivalent of said gram molar quantity of said metal hydride;

(d) passing the resultant solution of step (c) through a filter to remove precipitates of salt formed by said metal and said acid.

2. The method of claim 1 wherein said step (a) includes the step:

selecting said metal hydride to be calcium hydride and acid to be sulfuric acid.

3. Canceled.

4. A solution consisting essentially of less than 2500 parts per million of calcium sulfate, and a pH of less than 2.5 wherein pH of less than 2.5 maintained for longer than 48 hours.

5. A method for preparing a solution having a pH of less than 2.5, and less than 2500 parts per million of calcium sulfate which comprises the steps of:

(a) forming a solution of one mole H_2SO_4 per one liter of water;

(b) stirring into said solution of step (a) one Gram Equivalent Weight of CaH_2 per one mole of H_2SO_4;

(c) filtering the solution of step (b) through an eleven micron filter;

(d) allowing the solution of step (c) to digest for at least 10 hours;

(e) filtering the solution of step (d) through a two micron filter.

STATEMENT UNDER ARTICLE 19(1)

It is respectfully submitted that Applicant's claims are patentably distinct from the teaching of each of the cited references as discussed below:

A. CHEMICAL ABSTRACTS, vol. 88, no. 4, **does not teach**: (1) the solution consisting essentially of less than 2500 parts per million of calcium sulfate and a pH of less than 2.5; (2) using gram molar quantity of acid; (3) using equivalent of the gram molar quantity of the metal hydride; and (4) removing precipitates of salt formed by filtration.

B. THE MERCK INDEX, ELEVENTH EDITION also **does not teach**: (1) the solution consisting essentially of less than 2500 parts per million of calcium sulfate and a pH of less than 2.5; (2) using gram molar quantity of acid; (3) using equivalent of the gram molar quantity of the metal hydride; and (4) removing precipitates of salt formed by filtration.

C. US 5,830,838 **teaches** preparing the solution by mixing **calcium hydroxide and potassium hydroxide with equivalent sulfuric acid in water**. (*See*, Abstract and Col. 1, lines 53-67) It **does not** teach mixing gram molar quantity of an acid with an equivalent of the gram molar quantity of the metal hydride.

D. WO 94 /09798 A **teaches**: (1) isolation from natural materials, such as peat, using the extraction and purification procedures to obtain the inorganic compositions (*see*, page 3, lines 8-10), and (2) alternatively, producing by combining and/or synthesizing the constituent components, such as mixing an aqueous solution of potassium sulfate with an aqueous solution of calcium sulfate (*see*, page 10, lines 2-11). The reference **does not teach**: (1) the solution consisting essentially of less than 2500 parts per million of calcium sulfate and a pH of less than 2.5; (2) using gram molar quantity of acid; (3) using equivalent of the gram molar quantity of the metal hydride; and (4) removing precipitates of salt formed by filtration.

E. US 4,735,802 **does not teach**: (1) the solution consisting essentially of less than 2500 parts per million of calcium sulfate and a pH of less than 2.5; (2) using gram molar quantity of acid; (3) using equivalent of the gram molar quantity of the metal hydride; and (4) removing precipitates of salt formed by filtration.

F. US 5,895,782. Please see discussion of C. on US 5,830,838 above.

FIG. 1

1. select acid and metal hydride
2. add acid to water
3. add metal hydride to acid solution
4. filter the solution

FIG. 2

1. add one mole of concentrated sulfuric acid to water.
2. add 1 gram equivalent weight of analytic grade CaH_2 to the solution.
3. stir the solution.
4. pass solution through 10 micron filter.
5. allow solution to digest.
6. filter solution through a 2 micron filter.

INTERNATIONAL SEARCH REPORT

Intern ıal Application No
PCT/US 99/06247

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A01N59/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CHEMICAL ABSTRACTS, vol. 88, no. 4, 23 January 1978 (1978-01-23) Columbus, Ohio, US; abstract no. 26914, J.LELEU: "Dangerous chemical reactions. 43. Simple hydrides, boranes and silanes" XP002123421 abstract see IT "7664-93-9" (hazardous reactions with calcium hydride) & CAH. NOTES DOC., vol. 85, 1976, pages 583-589, --- -/--	1,2,5

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
22 November 1999	08/12/1999

Name and mailing address of the ISA	Authorized officer
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
X	S.BUDAVARI ET AL. (ED.): "THE MERCK INDEX, ELEVENTH EDITION" 1989 , MERCK & CO. , RAHWAY, N.J., US XP002123420 see MISC-104 - MISC-105, "Saturated Solutions"; "Calcium sulfate"	3,4
X	US 5 830 838 A (OVERTON JAMES M ET AL) 3 November 1998 (1998-11-03) column 1, line 39 - line 67	3,4
X	WO 94 09798 A (BONAPARTE CO) 11 May 1994 (1994-05-11) page 1, line 14 - line 16 page 2, line 17 - line 28 page 3, line 11 - line 23 page 4, line 29 -page 5, line 4 page 5, line 27 - line 31 page 10, line 29 - line 33 page 11, line 19 -page 12, line 7	1-5
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E	US 5 895 782 A (OVERTON JAMES MICHAEL ET AL) 20 April 1999 (1999-04-20) column 1, line 47 - line 53 column 2, line 15 - line 24	3,4

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INTERNATIONAL SEARCH REPORT

information on patent family members

Interr. ıal Application No
PCT/US 99/06247

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WO 9409798 A	11-05-1994	AU 5589594 A	24-05-1994
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